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STUDIES INTO THE THERMAL STABILITY AND REACTIVITY OF AMMONIUM NITRATE PART 1: THE REACTIVITY OF VARIOUS METALS WITH AMMONIUM NITRATE STUDIED BY ACCELERATING RATE CALORIMETRY

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STUDIES INTO THE THERMAL STABILITY AND REACTIVITY OF AMMONIUM NITRATE
PART 1: THE REACTIVITY OF VARIOUS METALS WITH AMMONIUM NITRATE STUDIED BY ACCELERATING RATE CALORIMETRY

by

P.P. Lee* and R.R. Vandebeek**

ABSTRACT

Thermal decomposition of ammonium nitrate-metal (Fe, Al, Ni, Pb, Cu, Cr, Zn) mixtures was studied by accelerating rate calorimetry.

The results showed that mixtures of ammonium nitrate with Pb, Cu, Zn, and Ni underwent reaction at a temperature below the melting point of ammonium nitrate with the formation of new compounds. Ammonium nitrate decomposed rapidly at 171°C in the presence of Cr. No significant effect from Fe and Al on the decomposition of ammonium nitrate was found.

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ÉTUDE PORTANT SUR LA STABILITÉ THERMIQUE ET LA RÉACTIVITÉ DU NITRATE D'AMMONOIUM

1IÈRE PARTIE: LA RÉACTIVITÉ DU DIVERS MÉTAUX AVEC LE NITRATE D'AMMONIUM ÉVALUÉE PAR CALORIMÉTRIE À RÉGIME ACCÉLÉRÉ

par

P.P. Lee* et R.R. Vandebeek**

RÉSUMÉ

La décomposition thermique de mélanges nitrate d'ammonium-métal (Fe, Al, Ni, Pb, Cu, Cr, Zn) fut évaluée par calorimètrie à régime accéléré.

Les résultats obtenus montrérent que les mélanges nitrate d'ammonium - Pb, Cu, Zn, Ni ont réagi à une température inférieure au point de fusion du nitrate d'ammonium, avec formation de nouveaux composés. Le nitrate d'ammonium se décomposa rapidement à 171°C, en présence de Cr. Fe et Al n'eurent aucune action significative sur la décomposition du nitrate d'ammonium.

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INTRODUCTION

Ammonium nitrate (AN) is a major ingredient in commercial explosives. The stability of ammonium nitrate has been studied very extensively for decades. However, most of the studies were performed at a temperature above the melting point. Some of the areas studied are summarized below:

1. phase transition study [1-3];
2. dehydration [4];
3. thermal decomposition and mechanism [5-8];
4. effect of gaseous products on the thermal decomposition [9-12];
5. the decomposition of AN in the presence of oxidizable materials, organic compounds and metallic contaminants [13-16];
6. thermal stability evaluation of AN by thermal analytical techniques, DSC and DTA [17-19]; and
7. investigation of the explosibility and properties [20-21].

The addition of foreign materials to ammonium nitrate can have different effects on the stability of the ammonium nitrate. A study by Rozman and Borodkina [14] shows that the addition of 0.03-0.1% urea reduces the decomposition of AN at 200°C and 225°C. Results from Findlay and Rosebourne [13] indicate that AN undergoes decomposition at a comparatively low temperature in the presence of woodmeal and starch. When the addition of such materials lowers the thermal stability of an explosive, these materials are termed thermally incompatible. If incompatible materials are present, the degree of hazard may change associated with the handling. In addition, the explosive system properties may alter during its storage. When normally inert materials are mixed with explosives, the properties of the mixture cannot be inferred from the properties of the components. Such incompatibility may be caused by secondary chemical reactions, mobility of solvents or gases.

Queen's University [16] worked on the decomposition of AN in the presence of metals using a rapid heating test involving 50 g samples. The mixtures of AN and metal, e.g., Pb, Cr, Cu, Zn, showed accelerated decomposition. However, no reaction mechanism was discussed. Because thermal instability of the mixture could lead to hazards in handling, manufacturing and storage, more studies were required.

An accelerating rate calorimeter (ARC) manufactured by Columbia Scientific Industries, Texas, has been used by the Canadian Explosives Research Laboratory for thermal stability research and incident investigation. The instrument has high sensitivity and may provide some information on the pressure data of the reactions. This report deals with the detected phenomena of decomposition of AN and metal mixtures by accelerating rate calorimetry. A few commonly used metals were used in this study because they could possibly be introduced as contaminants in the explosives.

EXPERIMENTAL

PROCEDURE

The design, operation and performance of the ARC have been published in the literature [22-25] and will not be discussed further.

All experiments were conducted in wide-mouth cylindrical Hastelloy C bombs weighing approximately 49 g each. Six stainless steel screws secure a thin metal disk and a Teflon gasket to a flange on the bomb. The bomb and its contents are suspended inside the calorimeter.

The weight of sample used in the experiment was determined based on previous experiments. A 1.5 g sample corresponding to a thermal inertia (ϕ) of 9 was used because it did not cause major damage to the calorimeter and was close to the ideal situation at which ϕ is equal to 1. Some reactions were too vigorous and the weight of sample was reduced.

The step heat method was used to study the reaction and decomposition of AN and metal mixtures. The first step was to heat the sample and container to the start temperature at 100°C. Then the calorimeter was allowed to stabilize for 30 min. After equilibrium, the self-heat rate was monitored at a threshold detection limit, usually 0.02°C/min, for an exotherm. When no exotherm was detected, the sample was heated 5°C. This heat-wait-search process was continued until a self-heat rate greater than 0.02°C/min was detected. After detection of the exotherm, time, temperature and self-heat rate were monitored until the end of the reaction.

Because the exothermic reaction was detected by ARC at a temperature below the melting point of AN for some mixtures, an isothermal method was employed to investigate the lowest temperature at which the exothermic reaction occurred. Basically, the sample was maintained adiabatically at a

given temperature for a certain period of time. If the self-heat rate greater than 0.02°C/min was detected during the aging, the system automatically went into the data acquisition mode. The sample was aged isothermally at a temperature at which the reaction could take place and that temperature was determined based on the result from step heat experiments. After the 600 min isothermal age period, the experiment was stopped at a certain temperature. The physical appearance of the contents was recorded. The weight loss and other properties, such as solubility in water, pH and UV absorbance, were studied briefly.

MATERIALS

1. Ammonium nitrate crystal, ACS grade, Anachemia Ltd.
2. Iron metal fillings, 40 mesh, Anachemia Ltd.
3. Aluminum scrap, for use in explosives, Ireco Co.
4. Aluminum powder, DuPont Co.
5. Nickel powder, 99.999%, Aldrich Chemical Co.
6. Lead granular, 20 mesh, ACS grade, Baker Chemical Co.
7. Copper powder, purified, Baker Chemical Co.
8. Chromium powder, 325 mesh, 99%, Aldrich Chemical Co.
9. Zinc granular, 30 mesh, ACS grade, Baker Chemical Co.
10. Zinc powder, purified, Baker Chemical Co.

Ammonium nitrate was stored in a desiccator before being used in these experiments. The AN-metal mixtures, in a ratio of 95:5 or 90:10, were prepared by mixing accurately the two components and storing in a desiccator. When the metal was in larger granular form, no homogeneous mixture could be made, hence only the 10% mixture was used for these metals.

RESULTS

STEP HEAT STUDIES

Thermal decomposition experiments were carried out at a start temperature of 100°C with a step heat cycle of 5°C and a wait time of 30 min. Decomposition of AN crystal was performed as a control. All the data are summarized in Table 1. The decomposition curves of the mixtures are shown in Fig. 1-8.

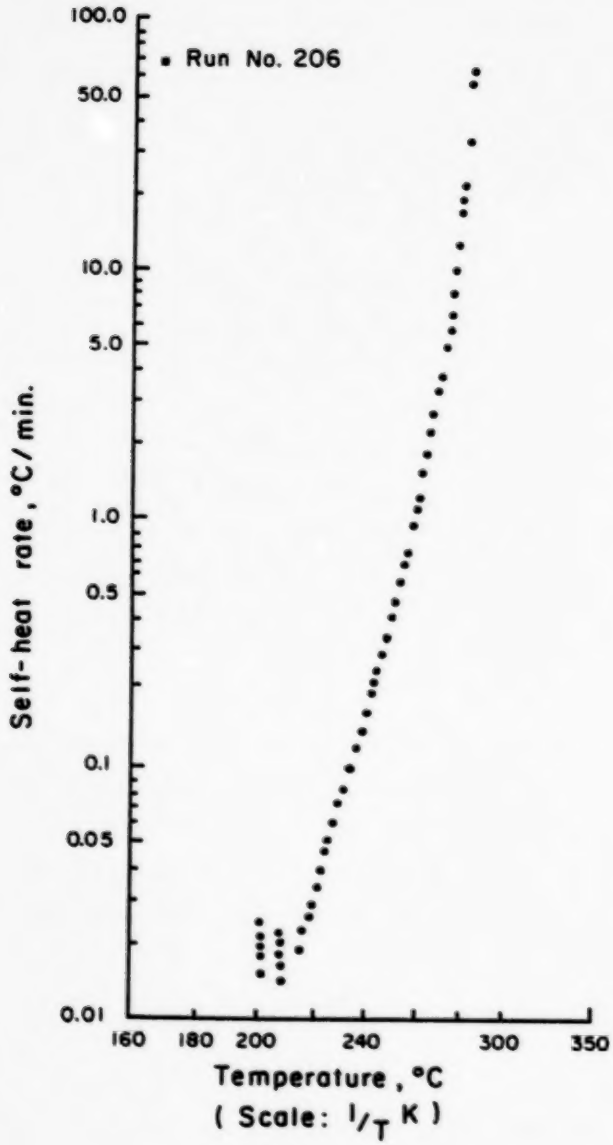

Fig. 1 - Thermal decomposition of ammonium nitrate crystal

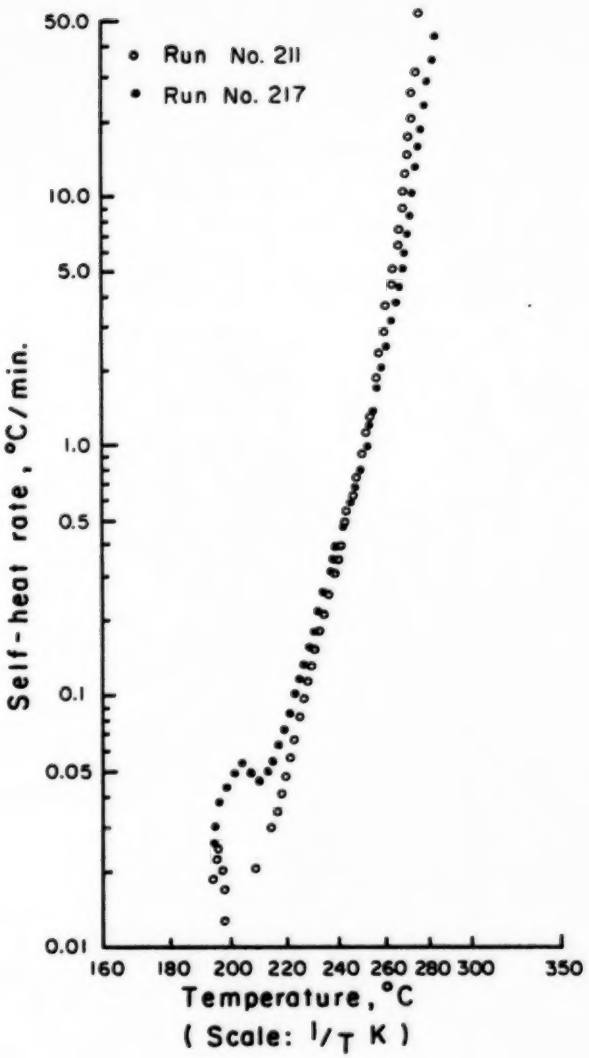

Fig. 2 - Thermal decomposition of ammonium nitrate crystal and 10% Fe, 40 mesh

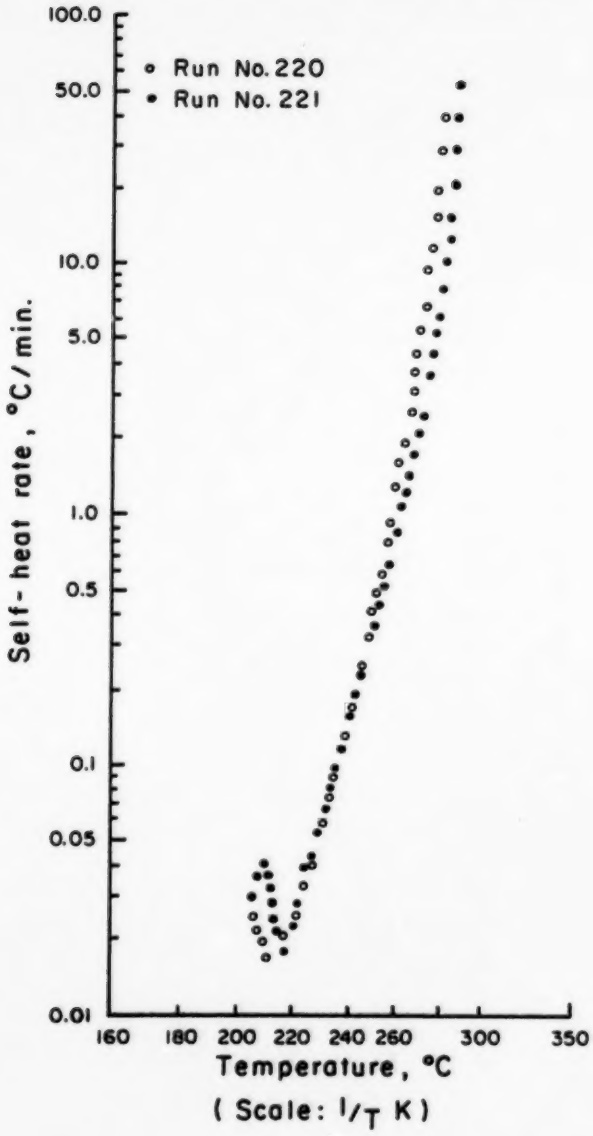

Fig. 3 - Thermal decomposition of ammonium nitrate and 5% Al powder

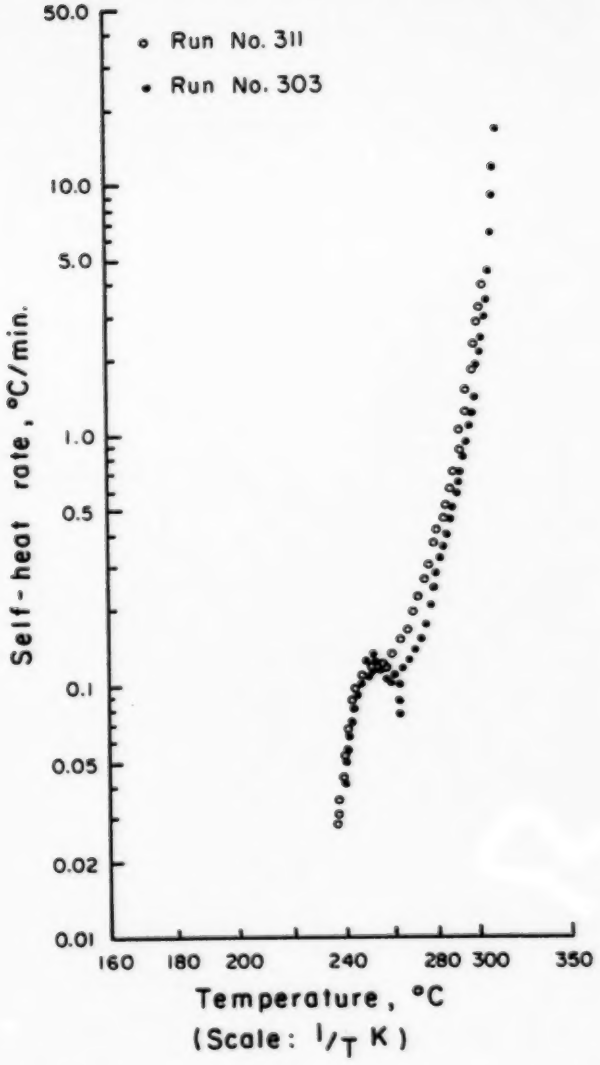

Fig. 4 - Thermal decomposition of ammonium nitrate crystal and 5% Ni powder

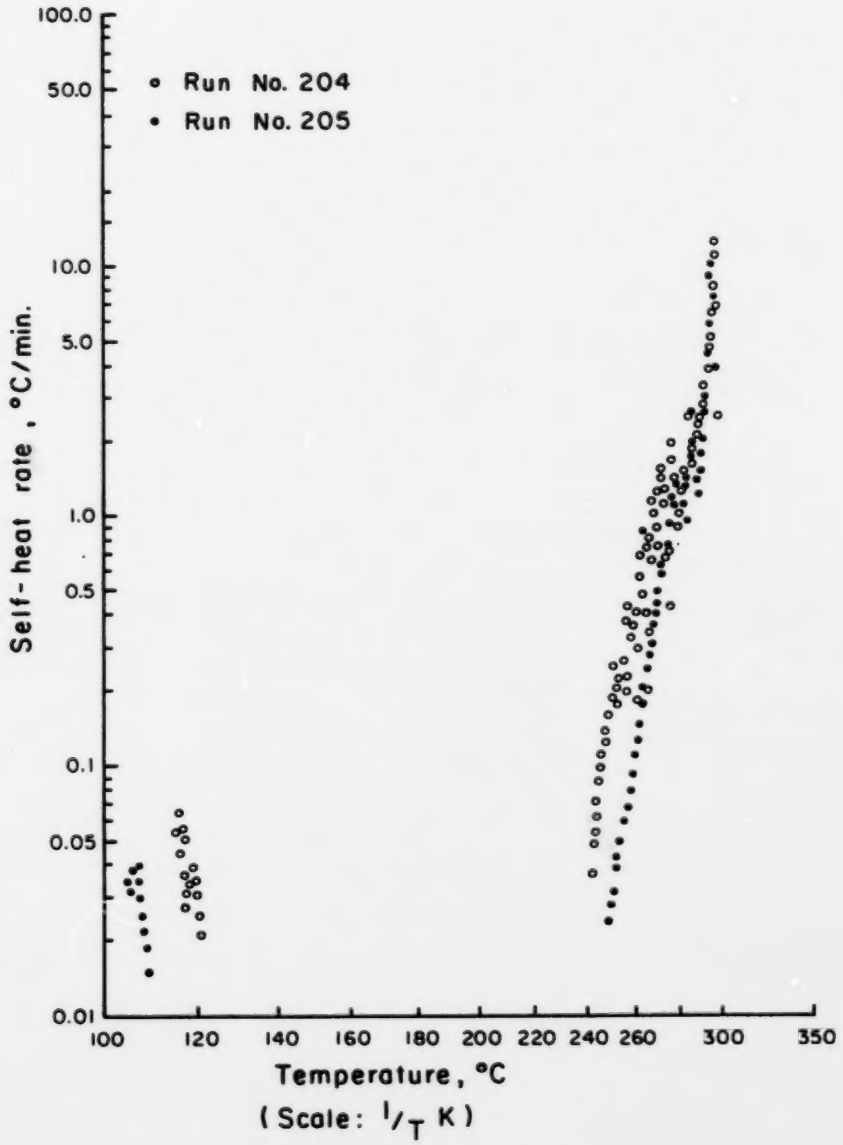

Fig. 5 - Thermal decomposition of ammonium nitrate crystal and 10% Pb, 20 mesh

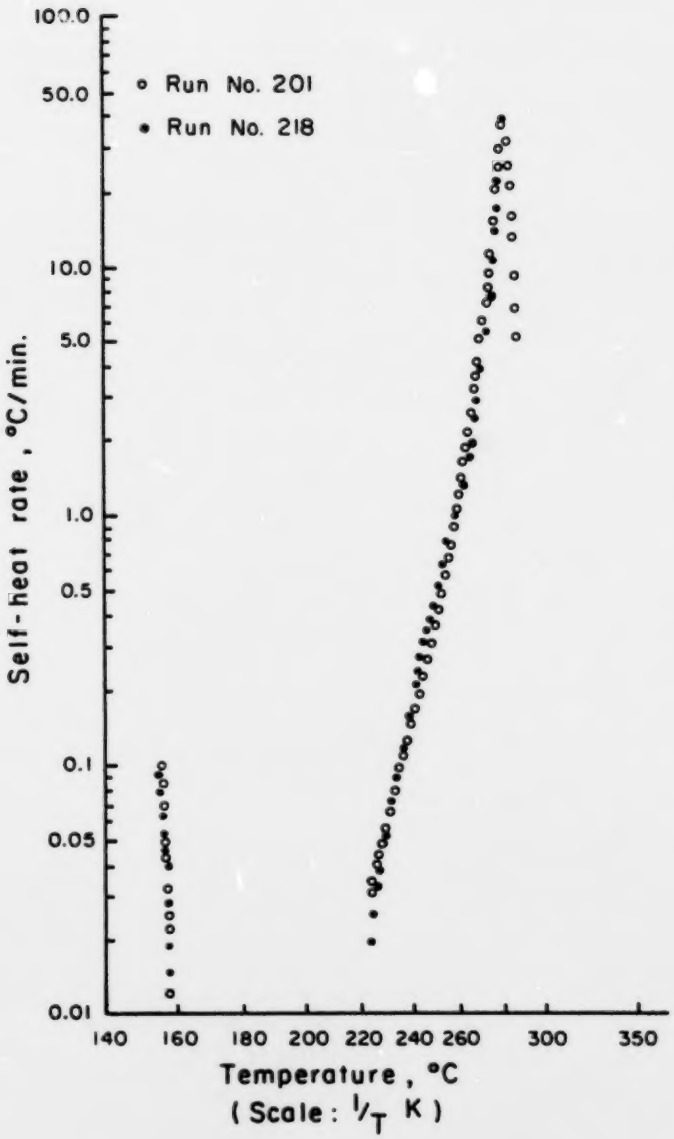

Fig. 6 - Thermal decomposition of ammonium nitrate crystal and 5% Cu powder

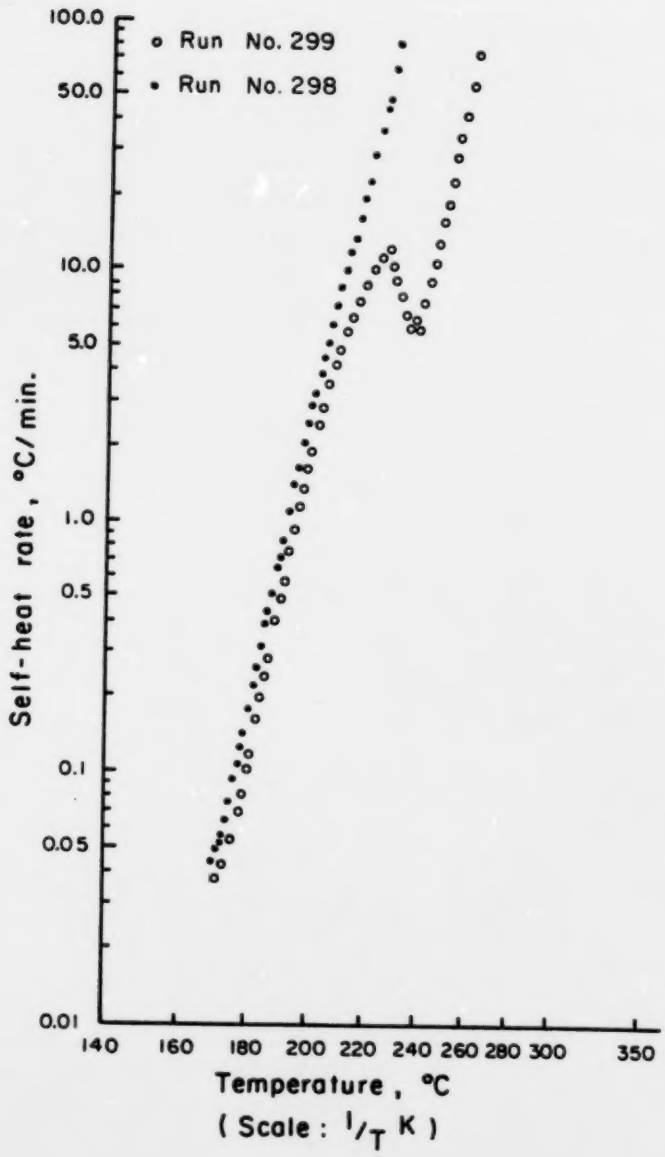

Fig. 7 - Thermal decomposition of ammonium nitrate crystal and 5% Cr powder

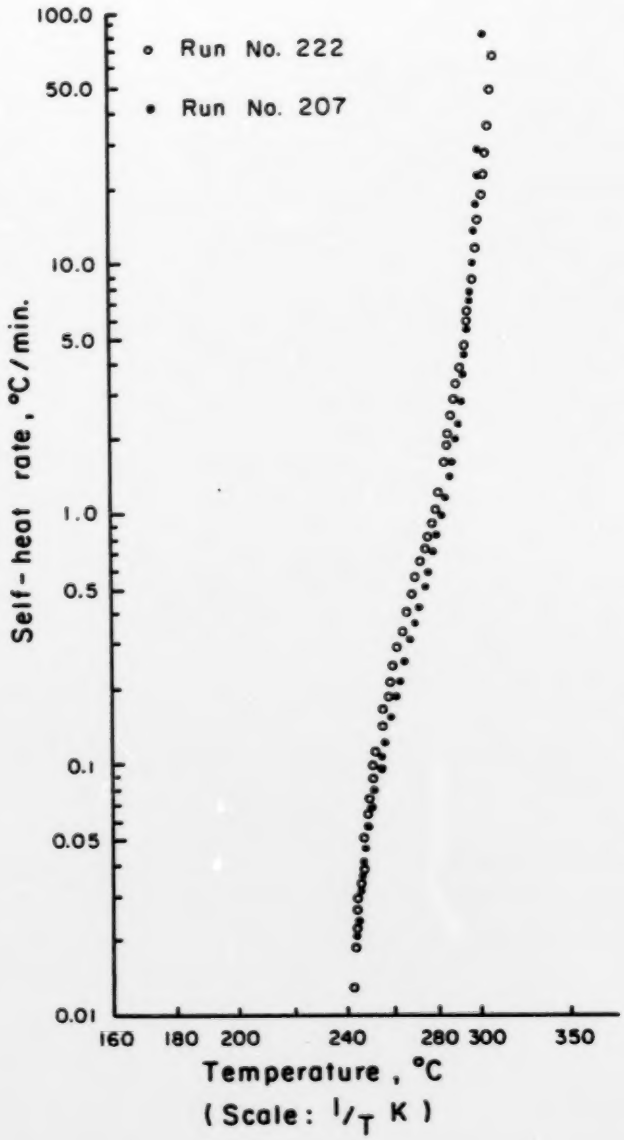

Fig. 8 - Thermal decomposition of ammonium nitrate crystal and 10% Zn, 30 mesh

Table 1 - Thermal decomposition data of AN-metal mixtures

Run No.	φ	1st exotherm T (°C)	1st exotherm m (°C/min)	Exotherm leading to ignition: Initial data T_o (°C)	Initial data m_o (°C/min)	Before ignition T (°C)	Before ignition m (°C/min)	At maximum rate T_m (°C)	At maximum rate m_m (°C/min)	Wt loss (%)
AN crystal										
185	8.6	201.35	0.02	210.13	0.026	262.20	2.157	292.28	117.285	98.8
206	9.0	201.36	0.02	215.46	0.022	262.54	2.050	286.73	112.666	98.1
AN + 10% Fe, 40 mesh, Anachemia										
211	9.1	196.69	0.02	216.85	0.033	258.38	2.000	283.07	117.833	87.5
217	9.0	196.39	0.02-0.05	214.40	0.051	260.07	2.000	287.71	117.000	87.8
AN + 5% Al scrap, Ireco										
199	9.0	191.23	0.02	198.86	0.026	259.05	2.166	276.04	80.000	92.8
208	9.0	186.31	0.02	194.93	0.023	256.04	2.129	281.33	125.500	93.1
AN + 5% Al powder, DuPont										
220	9.0	206.29	0.02	217.25	0.023	266.31	1.952	299.43	184.333	92.5
221	8.9	206.33	0.02	217.35	0.023	272.84	2.000	303.02	116.666	93.4
AN + 5% Ni powder, Aldrich										
303	10.2	140-142	fast	241.71	0.047	303.25	2.050	314.84	53.000	88.9
311	9.1	140-142	fast	238.27	0.031	297.96	2.033	301.76	stop	93.4
AN + 10% Pb, 20 mesh, Baker										
204	9.0	116-122	0.02-0.05	243.63	0.050	288.80	1.939	298.95	12.000	77.3
205	8.9	107-111	0.02-0.04	251.88	0.026	288.49	2.470	297.12	10.000	75.9
AN + 5% Cu powder, Baker										
201*	8.9	157-159	0.02-0.09	225.36	0.032	262.94	2.000	279.41	39.000	100.0
218	13.0	156-159	0.02-0.05	225.21	0.026	266.73	2.157	286.33	100.500	90.3
AN + 5% Cr powder, Aldrich										
298*	9.6	-	-	171.93	0.047	198.52	2.000	231.22	82.000	-
299	13.9	-	-	171.75	0.037	201.80	2.050	227.92	12.500 (slow down)	
								272.85	153.333	-
313	13.2	-	-	171.48	0.031	201.59	1.935	229.64	11.750 (slow down)	-
								269.74	110.000	91.4
AN + 10% Zn, 30 mesh, Baker										
207	8.9	135-177	Very fast	246.21	0.022	289.25	2.000	304.82	82.500	83.9
222	8.8	135-168	Very fast	244.56	0.020	285.74	1.937	316.48	100.833	89.1
AN + 5% Zn powder, Baker										
314	13.3	-	-	241.25	0.024	293.39	2.103	299.84	5.000	90.2
AN + 10% Zn powder, Baker										
315	12.8	-	-	246.15	0.025	293.72	2.000	306.78	93.000	84.3

* experiments with ruptured disks

AN + 10% Fe, 40 mesh

The AN-Fe mixtures were found to have similar decomposition patterns to that of pure AN crystal. A slight exotherm took place at 196°C. The major exotherm started at approximately 215°C with the temperature just before ignition being 260°C.

AN + 5% Al scrap, Ireco Co.

The mixture of AN and Al scrap from Ireco Co. was determined to have a major exotherm starting at a temperature of 198°C. However, the temperature before ignition occurred in the same region as the AN-Fe mixture.

AN + 5% Al powder, DuPont Co.

The first exothermic reaction was detected at 206°C with a very slow self-heat rate of 0.02°C/min. The major exotherm leading to ignition commenced at 217°C. An ignition occurred at 300°C.

AN + 5% Ni powder

An abrupt temperature rise of 2°C at 140°C was detected. It indicated that a fast exothermic reaction took place and this phenomenon was observed in duplicate runs. The major exotherm commenced at 240°C, which is about 30°C higher than for pure AN crystal. These results suggest that a new compound was formed at 140°C and that was stable to 240°C. Temperature before ignition was 300°C compared to 262°C for pure AN. The decomposition of the compound was not a smooth pattern as evident from Fig. 4. It shows a complex decomposition with a shoulder followed by a fast self-heat rate and ignition.

AN + 10% Pb, 20 mesh

When the mixture was tested by ARC, a slight exotherm at 110°C was detected with a slow self-heat rate at 0.02-0.04°C/min, and the total temperature rise was 4°C. The material was then stable to 250°C, at which point the major exotherm started. During the course of decomposition, the self-heat rate fluctuated around 1-2°C/min for a period of time and then ignited. The above observations strongly indicate that a new compound was formed. Because an exotherm occurred at such a low temperature, it is possible to initiate a thermal process and the reaction has to be examined closely. Figure 5 shows the results of duplicate test of the AN and Pb mixture.

AN + 5% Cu powder

Duplicate tests of this mixture gave very reproducible results. The first short exotherm was detected at 156°C with slow self-heat rate and total temperature rise of about 3°C. Then the sample was stable to 225°C, at which point the major exotherm commenced. Again it indicated the new compound was formed during the first exothermic reaction. The ignition of the material was very vigorous and the sealing disk rated for 6.9 MPa ruptured.

AN + 5% Cr powder

This mixture did not show any exothermic reaction below the melting point of the AN. The exothermic decomposition started at 171°C and accelerated very rapidly. The temperature before ignition was in the 201°C range. At a test with ϕ equal to 9.6, explosion occurred with rupture of the disk. Therefore, the sample size was reduced in later experiments resulting in a ϕ factor of 13. The decomposition curve shows a two-step reaction (Fig. 7).

AN + 10% Zn, 30 mesh

The mixture reacted extremely rapidly at 135°C with a temperature jump to 170°C. The instrument was unable to trace the process and the temperature increase was only shown in the data print out. The sample was stable until the major exotherm started at 246°C. It is suspected that a new compound was formed during the first exotherm. It started to decompose at 245°C and subsequently ignited vigorously. The second exotherm, self-heat rate as a function of $1/T$, is plotted in Fig. 8. Because the temperature increased 35°C abruptly at 135°C, a large amount of energy was released and it could introduce a significant thermal hazard.

AN + 10% Zn powder

AN and Zn powder mixtures did not show the fast temperature increase at 135°C. The material was stable to 246°C before the exotherm was detected and the decomposition curve was similar to the mixture of AN-Zn, 30 mesh. The reasons for this difference are not apparent.

ISOTHERMAL AGE STUDIES

Based on the results of thermal decomposition of the AN-metal mixtures obtained using the step heat method, several mixtures showed that

chemical reactions took place at a temperature below the melting point of the AN. An investigation into the lowest temperature at which the reaction occurs and the possibility of new product formation is desirable. Consequently, the mixtures were isothermally aged at a pre-determined temperature for 600 min and step heated to a certain temperature. After the test, the phenomena detected by ARC, the physical appearance of the sample, weight loss, and some properties of the final products are reported. The UV-visible spectra of the reacted material shows the presence of nitrate at 302 nm. All the experimental data are summarized in Table 2.

AN + 10% Fe, 40 mesh

AN-Fe mixtures were isothermally aged at 160°C for 600 min. Unreacted metal was found and the weight loss was 0.5%. No pressure build-up was shown on the sealing disk. After dissolving the material in water, the solution appeared slightly yellow with dark brown particles.

AN + 5% Al, scrap, Ireco Co.

After the mixture was aged at 160°C for 600 min, the AN crystals were partially melted. The water solution was light yellow with metal particles present.

AN + 5% Ni powder

No reaction in the 120°C to 140°C range was detected. When the mixture was aged at 140°C and stopped at 160°C, a slight exotherm was detected at 144°C. The final product was bluish green in colour and was quite soluble in water.

AN + 10% Pb, 20 mesh

The AN and Pb mixture was isothermally aged at 100°C for 600 min and stopped at 120°C. After reaction, a white molten material was formed, Pb granules disappeared and the weight loss was 2.2%. The material formed by the reaction was quite soluble in water with a pH of 7-8.

AN + 5% Cu powder

Cu powder reacted with AN at 156°C as shown by the step heat method. The AN-Cu mixture was aged at 150°C for 600 min and ended at 160°C. The

Table 2 - Isothermal age studies on AN-metal mixtures

Run No.	Sample	ϕ	Isothermal age: Start temp (°C)	Isothermal age: Time (min)	Isothermal age: End temp (°C)	Phenomena detected by ARC	Physical appearance	Properties	Wt loss (%)
216	AN crystal	9.0	160	600	165	Very stable isothermal age for 600 min; no pressure build-up shown at the sealing disk	AN crystals stick together	Soluble in water	0.3
215	AN + 10% Fe, 40 mesh	9.0	160	600	165	Same as above	AN crystals stick together with Fe present	Water solution was slightly yellow with dark brown particles	0.5
214	AN + 5% Al, scrap	9.0	160	600	165	Same as above	AN partially melted with Al present	Water solution was slightly yellow with metal particle; pH 6	0.4
296	AN + 5% Ni powder	8.9	120	600	140	Stable isothermal age at 120°C for 600 min	AN stick together with Ni present	Soluble in water with metal present	-
297	AN + 5% Ni powder	9.1	140	600	160	Slight exotherm occurred at 144°C; then stable for 600 min	Bluish-green molten material	Quite soluble in water; greenish solution	0.5
213	AN + 10% Pb, 20 mesh	8.8	100	600	120	Temperature increased to 102°C and remained stable isothermal age for 600 min	White molten material with no metal present	Soluble in water; pH 7-8	2.2
212	AN + 5% Cu powder	9.0	150	600	160	Slight temperature increase then remained stable at isothermal temperature for 600 min	Blue molten material with no metal present	Blue water solution with white residue; pH 7; UV-VIS absorption at 302 nm and 640 nm	0.9

Table 2 (Cont'd)

Run No.	Sample	ϕ	Isothermal age: Start temp (°C)	Isothermal age: Time (min)	Isothermal age: End temp (°C)	Phenomena detected by ARC	Physical appearance	Properties	Wt loss (%)
302	AN + 5% Cr powder	13.8	160	600	180	Stable isothermal age at 160°C for 600 min; exotherm occurred at 166°C	Yellow molten solid with black particles	Orange-yellow water solution with black precipitate	6.6
312	AN + 5% Cr powder	13.8	160	1440	166	Stable age at 160°C for 1440 min; exotherm occurred at 164.5°C	Same as above	Same as above	2.5
210	AN + 10% Zn, 30 mesh	9.0	130	600	190	Temperature jumped from 130°C to 181°C; then isothermal age was stable for 600 min at 181°C; pressure build-up was shown	white molten material with no metal present	slightly soluble in water; pH 7	8.2
209	AN + 10% Zn, 30 mesh	9.0	130	1440	320	Temperature jumped from 130°C to 188°C; the main exotherm commenced at 248.5°C	--	--	84.3

resulting material was blue in colour with no metal present. After dissolving the product in water, the solution was blue with white residue. The UV-VIS absorption peaks were at 302 nm and 640 nm.

AN + 5% Cr powder

From the results of step heat runs, Cr powder reacts exothermically with AN at 171°C and ignited more vigorously than AN crystal. An isothermal age experiment for AN-Cr mixture was performed at 160°C for 1440 min and the material survived the aging. An exotherm was detected at 165°C with a yellow molten material and black particles as final products.

AN + 10% Zn, 30 mesh

An abrupt reaction occurred at 135°C for the reaction of the AN-Zn mixture as reported in the previous section of this report. The results from the isothermal aging experiment demonstrated that the temperature jumped 50°C immediately. After reaction, a white molten material with no metallic Zn was observed. Pressure build-up was shown at the sealing disk and the weight loss was 8.2%.

DISCUSSION

Very few investigations on decomposition of ammonium nitrate below its melting point have been reported in the literature. An article was cited in a Russian journal, discussing this matter by Moshkovich and co-worker [26]. The study on the reaction of AN and metal mixtures are rarely found in the literature. Japanese workers Soda and Endo [27] reported a study on explosibility of AN-metal powder systems using an impact sensitivity test and the Krupp ignition test. Reactions of AN-metal mixtures were only studied very briefly by Queen's University [16].

In these experiments, some valuable information has been identified. The AN-metal mixture may react violently at a temperature below the melting point of the AN. The experimental results show that their mixtures reacted in the solid state and formed new compounds. The reactions are dependent on many variables, e.g., metal size, purity of metal, the ratio of the mixture, the confinement and the experimental system. The reaction products are also mixtures, and their characteristics will be reported in the future.

In some of the studied reactions, the metals reacted with the AN below its melting point, others just above its melting point and finally some did not appear to affect the normal thermal decomposition pattern of the AN. Some of the reaction products formed, e.g., by Cu and Cr, were sufficiently energetic to burst the rupture disks used. As discussed by Garner [28], in solid reactions there are many complications. The decomposition is affected by the rate of nuclei formation. Also, the solid may crack under the strains created by the formation of the new solid phase. Small liquid inclusions may give rise to abnormalities. When very small particles are used, sintering may take place during the solid decomposition. Our experiments, involving the reactions of the mixture followed by decomposition, lead to a very complicated situation. In this paper attention is brought to the observations and the phenomena detected by ARC during the course of each reaction. Insufficient data are available to discuss the mechanism of the reactions.

CONCLUSIONS

Thermal hazards of some AN-metal mixtures have been evaluated. The results showed that Ni, Pb, Cu, and Zn reacted with AN at temperatures below the melting point of the AN. The heat evolved may trigger further reaction and may cause instability of the material. Cr reacted with AN rapidly at 171°C. However, Fe and Al did not affect the decomposition of AN significantly.

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